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Note

High-performance liquid chromatography of decamethrin

D. MOUROT, B. DELÉPINE, J. BOISSEAU and G. GAYOT
Laboratoire National des Médicaments Vétérinaires, Javené, 35300 Fougères (France)
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Decamethrin (DECIS, NRDC 161), (*S*)-α-cyano-3-phenoxybenzyl-*cis*-(1*R*,3*R*)-2,2-dimethyl-3-(2,2-dibromovinyl)cyclopropane carboxylate is a newly developed pyrethroid insecticide; it is the most potent insecticide currently known[1].

Ultraviolet techniques cannot be used directly because decamethrin exhibits an absorption maximum at 230 nm and many other organic substances present in the formulations absorb strongly at this wavelength. The separation of decamethrin from its photolysis degradation products using thin-layer chromatography or gas–liquid chromatography has been reported[2], but has not been applied to quantitation of the active constituent.

High-performance liquid chromatography (HPLC) offers an alternative for the analysis of pyrethroid insecticides[3,4] and this paper describes the HPLC analysis of decamethrin in reversed- and normal-phase modes.

EXPERIMENTAL

Chromatographic system

A Varian LC 8500 high-pressure liquid chromatograph with a variable-wavelength UV detector operating at 230 nm was used. The two chromatographic columns were a LiChrosorb RP-8 (Merck, Darmstadt, G.F.R.) column (10 cm × 4.7 mm I.D.), particle size 10 μm, and a LiChrosorb Si-60 (Merck) column (15 cm × 4.7 mm I.D.), particle size 5 μm. The flow-rates were maintained at 70 and 80 ml/h, respectively (column pressure 500 p.s.i.).

Chemicals

The reference sample of decamethrin was a generous gift from Roussel Uclaf–Procida (Paris, France). All solvents were of analytical-reagent grade and *n*-hexane and diisopropyl ether were anhydrous materials.

Analytical method

In the normal-phase system, isocratic elution was carried out with *n*-hexane–diisopropyl ether (93:7); for the RP-8 column, isocratic elution was carried out with acetonitrile–1% sulphuric acid (70:30).

RESULTS AND DISCUSSION

Decamethrin, being a well defined optical isomer of a group of eight possible isomers, appears as a single peak in both reversed- and normal-phase systems.

On the Si-60 column (see Fig. 1a), the order of elution was internal standard (diphenylamine) and decamethrin, the retention times being 4.5 and 7.6 min, respectively. However, replicate injections of decamethrin led to decreasing retention times, as described previously for sumicidin[4]. Pumping of pure diisopropyl ether through the column for 2 min followed by stabilization was also necessary in order to obtain good reproducibility of retention times.

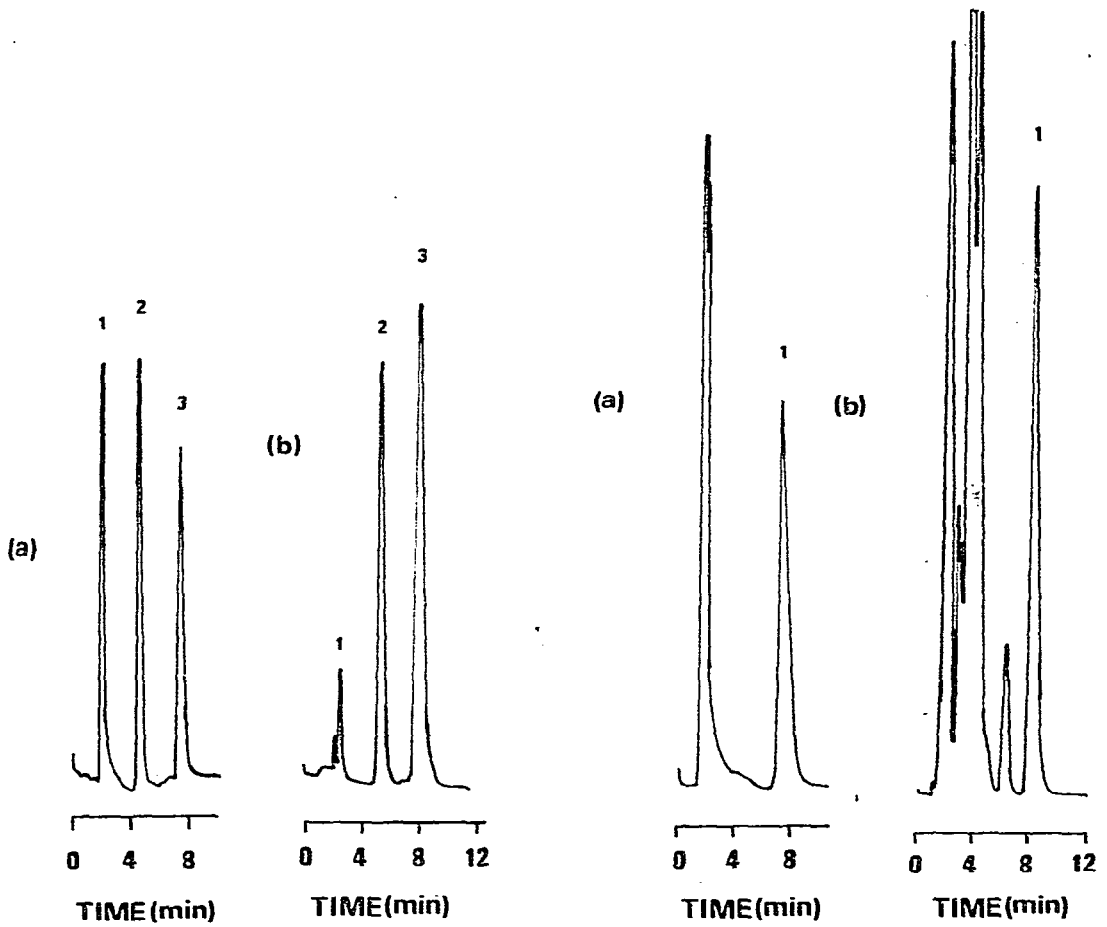

Fig. 1. HPLC of decamethrin in reversed- and normal-phase modes. (a) LiChrosorb Si-60 column; mobile phase, *n*-hexane–diisopropyl ether (93:7); flow-rate, 80 ml/h; detector sensitivity, 0.5 a.u.f.s. Peaks: 1 = injection artefact; 2 = diphenylamine (internal standard); 3 = decamethrin (0.5 g/l in chloroform). (b) LiChrosorb RP-8 column; mobile phase, acetonitrile–1% sulphuric acid (70:30); flow-rate, 70 ml/h; detector sensitivity, 0.5 a.u.f.s. Peaks: 1 = injection artefact; 2 = pentachlorobenzene (internal standard); 3 = decamethrin (1 g/l in methanol).

Fig. 2. HPLC scan of decamethrin formulation in reversed- and normal-phase modes. (a) LiChrosorb Si 60 column; (b) LiChrosorb RP-8 column. Conditions as in Fig. 1. Peak 1 = decamethrin.

However, in the reversed-phase system, excellent reproducibility of retention times was attained and, as shown in Fig. 1b, the order of elution was pentachlorobenzene (internal standard) and decamethrin, with retention times of 5.3 and 7.7 min, respectively.

Fig. 2 shows the chromatograms of a decamethrin formulation in reversed- and normal-phase modes. In each instance, the decamethrin peak was well defined without any significant interference encountered with other substances present in the formulation; however, on the RP-8 column, the internal standard peak was overlapped by a substance present in this insecticidal preparation. By modification of the acetonitrile to sulphuric acid ratio (*ca.* 60:40) and the flow-rate (*ca.* 90 ml/h) a good separation was achieved and quantitation by the internal standard method was still possible. Another formulation tested (as a powder) was quantitated according to the initial conditions.

CONCLUSION

This simple, rapid separation (less than 10 min) allows the presence of decamethrin in commercially available insecticidal preparations to be ascertained. It appears that both methods give excellent results and that the analysis is not affected by the presence of other substances if certain precautions are taken. The choice between the two is dependent on the composition of the formulation: oily-based formulations on the Si-60 column and aqueous formulations on the RP-8 column.

The application of this HPLC method to develop a screening test for the identification of pyrethrin or pyrethroid insecticide residues is now in progress and will be described in a subsequent publication.

REFERENCES

1 M. Elliot, A. W. Farnham, N. F. Janes, P. H. Needham and D. A. Pulman, *Pestic. Sci.*, 6 (1975) 537.
2 L. O. Ruzo, R. L. Holmstead and J. E. Casida, *J. Agr. Food Chem.*, 6 (1977) 1385.
3 E. J. Kikta, Jr. and J. P. Shierling, *J. Chromatogr.*, 150 (1978) 229.
4 D. Mourot, B. Delépine, J. Boisseau and G. Gayot, *J. Chromatogr.*, 168 (1979) 277.